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Nitrile rubber was used as the basis for developing a damper for the TR-317R tail mass assembly. The frequency of the maximum in loss tangent for nitrile rubbers was shown to depend strongly on acrylonitrile content of the polymer and, to a lesser extent, on the carbon black. Details of the application of the formulation that provides the best performance are given.

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DEVELOPMENT OF A NITRILE RUBBER FOR DYNAMIC DAMPING by

Corley M. Thompson.

Acoustical Materials Branch
Underwater Sound Reference Detachment
P.O. Box 568337
Orlando, Florida 32856-8337

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DEVELOPMENT OF A NITRILE RUBBER FOR DYNAMIC DAMPING

INTRODUCTION

The designers of the tail mass/spring arrangement for the TR-317 transducer recognized that there needed to be a loss mechanism (or damping) in that assembly in order to balance the losses that are produced at the front end of the transducer by the head mass rubber. They attempted a number of formulations before they found a proprietary butyl rubber that did an excellent job of balancing the transducer. Unfortunately, the rubber was specified by the rubber manufacturer by means of an ASTM specification intended for automotive uses. This specification did nothing to assure that the rubber had appropriate dynamic properties although the selected formulation worked well as long as it was purchased from that single supplier. When the design specification team for the TR-317 "R" transducer addressed this tail mass rubber, it was clear that the formulation being used was indeed proprietary and, thus, was not acceptable. A task was begun at the USRD to develop a non-proprietary rubber for the TR-317R transducer. This task focussed on nitrile rubber systems for three reasons: (1) there was more Navy experience with these; (2) nitrile rubbers have a more controllable loss since the basic polymer can be readily changed; (3) nitrile rubber, in general, has better engineering properties such as cure rate, strength, and bondability, than does butyl rubber.

Nitrile rubber is called by a variety of other names; among these are "Buna N," "NBR," several brand names (e.g. "Hycar" and "Paracril"), as well as the proper "acrylonitrile-butadiene copolymer rubber." It is the copolymer aspect of the last name that gives nitrile rubber its greatest design flexibility as a dynamic absorber. The polymer can be acquired with a wide range of acrylonitrile to butadiene ratios. Expressed as percent of acrylonitrile, these range from 18% to 45%. Work previously done at the USRD on another dynamic application of a nitrile containing 39% acrylonitrile had implied that this had a maximum in its loss at a frequency lower than the operational frequency of the TR-317R. There are several ways that might be considered as a means to change the frequency of the loss maximum. Among these are dilution of the nitrile polymer with a polymer that has a loss maximum at a higher frequency and adjustment of the percentage of acrylonitrile in the nitrile polymer. When the best match to the proprietary butyl rubber is achieved, then that formulation will be molded into TR-317 tail masses and tested before being specified for this use.

The literature on the dynamic properties of nitriles is somewhat sparse, particularly for frequencies in the audio range. On the other hand there is a great deal of information on the glass transition temperature of nitriles [1,2,3,4] and this may be useful since this is usually taken as the temperature of maximum loss. There is some published data on the dynamic properties of these elastomers at low frequencies -- typically from 1 to 100 Hz [4,5]. For higher frequency applications the usefulness of the low-frequency data is restricted to demonstrating trends. An early report

addressed the damping characteristics at frequencies of 25 Hz to 5 kHz for NBR's with a range of acrylonitrile contents [6]. These results show some trends in the loss of the rubber with different acrylonitrile contents, but do not show any maxima in loss tangent within this frequency range. In another report with a limited distribution, Madigosky and Satinover [7] give dynamic Young's modulus data for NBR's with two different acrylonitrile contents. Their data at 10°C show that a nitrile rubber with 39% acrylonitrile has a loss tangent maximum at 4 kHz, whereas a nitrile rubber with 26% acrylonitrile has a loss tangent maximum at greater than 100 kHz. They present data on elastomers based on other polymers but these are somewhat confounded by differences in sample compounding.

EXPERIMENTAL

Samples were prepared in a laboratory-size Banbury mixer by the Technical Sales and Engineering Co. of St. Petersburg, FL. The formulations of the samples were:

Polymer, as indicated	100 parts by weight
Zinc Oxide	5
Stearic Acid	1
Octylated Diphenylamine (ODPA)	2
Tetramethyl Thiuram Monosulfide (TMTM)	0.5
Sulfur	1.5
N550 Carbon black	40 to 80 parts, as indicated

The acrylonitrile butadiene polymers used were from the "Paracril" series by Uniroyal Chemical Division. The acrylonitrile contents were those reported by Uniroyal. Each sample was molded at 155°C to a cure state equivalent to 90% of the final cure as determined on a Monsanto R100S rheometer.

Dynamic mechanical properties of each formulation were determined on 0.63X0.63X7.5-cm strings using an apparatus developed at the USRD [8]. Samples were attached with cyanoacrylate adhesive to a Brüel and Kjaer Model 4810 Mini-Shaker. Brüel and Kjaer type 4374 accelerometers were attached to the ends of each sample. At each temperature a frequency sweep was applied to the shaker. The ratios of end accelerations at several resonant frequencies were measured from the accelerometers at each temperature. Measurements were made for each sample over a 0°C- to 50°C-temperature range at approximately 3°C intervals. The samples were allowed to equilibrate for approximately 0.5 hr at each temperature before they were measured. The end acceleration ratios were put into the solutions of the equations of motion to generate the modulus and loss tangent at each temperature and frequency. Frequency shift factors were applied to generate shifted modulus and loss tangent curves vs frequency at a reference temperature -- for this report, 10°C was chosen. These data may be shifted to some other temperature by means of an Arrhenius equation:

$$a_t = \exp\left[\frac{1}{T} - \frac{1}{T_{ref}}\right]^A , \qquad (1)$$

where a_t is the frequency shift factor, A is the Arrhenius constant, and the T's are in °K. The calculated frequency shift factor is simply multiplied by

the frequency to convert the data given to another temperature. In addition, the modulus must be corrected by multiplying by the ratio of the reference temperature to the target temperature.

RESULTS AND DISCUSSION

Design of Tail Mass Assembly Damper

The first step in attempting to match the dynamic performance of the butyl rubber was to obtain a sample for study. This was accomplished, with some difficulty, by acquiring a "string" of this material appropriate for measurement of its dynamic Young's modulus. This measurement was made and these data are given graphically as Fig. 1, below.
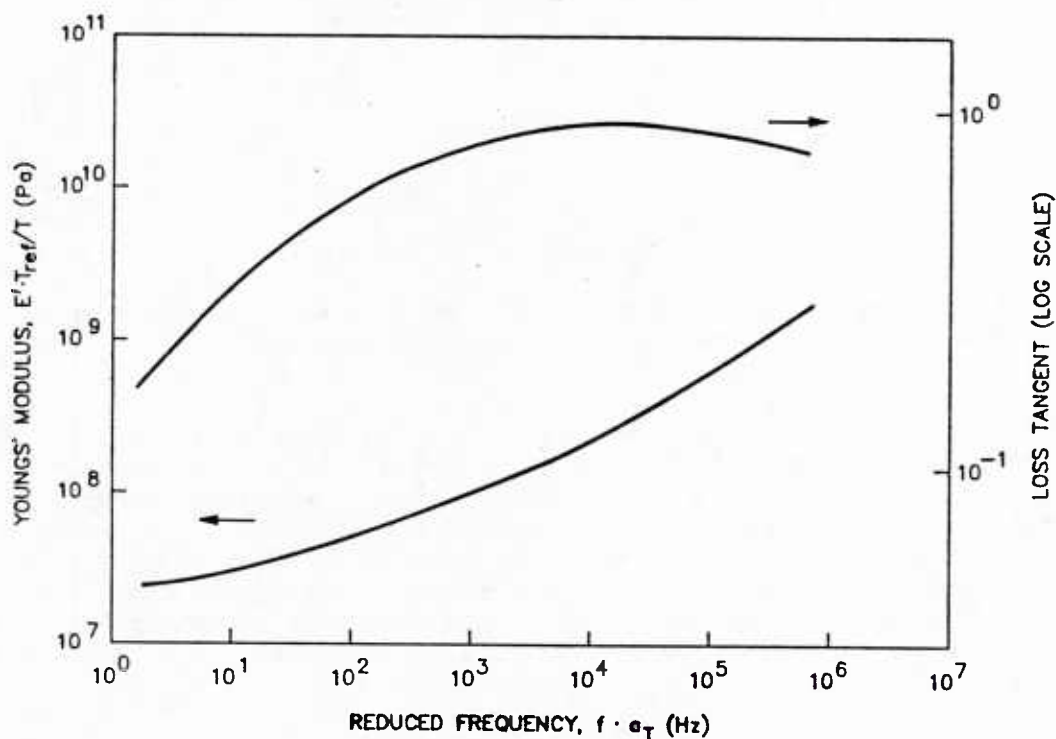

Fig. 1 – Dynamic Young's modulus and loss tangent for the proprietary butyl rubber formulation at a reference temperature of 15°C. The Arrhenius constant for this rubber is 5646 cal/mole.

The Navy's research community has had a great deal of experience with a formulation prepared with 39%-acrylonitrile polymer and filled with 50 parts of N550 carbon black. This was designated Nitrile 6100 and has been used for other shipboard damping applications. Its dynamic Young's modulus is shown in Fig. 2.
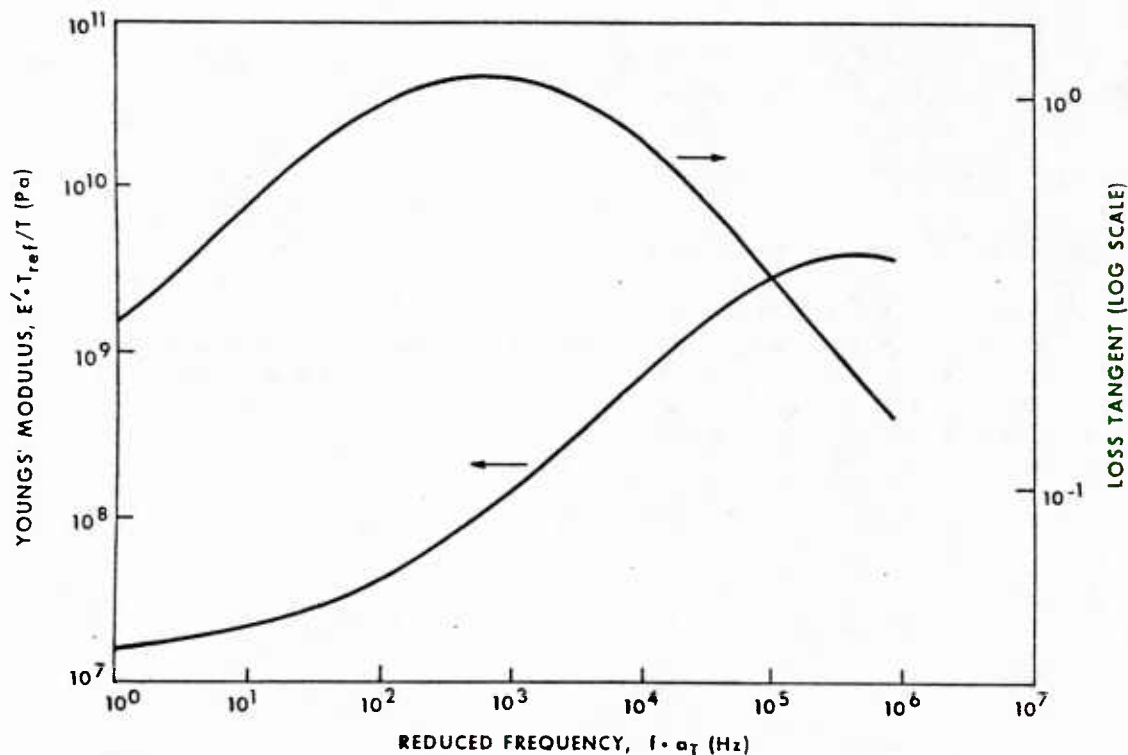
Fig. 2 - Dynamic Young's modulus and loss tangent for a 39%-acrylonitrile, 50-part N550 black nitrile rubber formulation. The Arrhenius constant is 6104 cal/mole.

This material has a loss-tangent peak that is lower in frequency than the proprietary butyl "target." Thus it was necessary to develop a method of raising the frequency of the loss tangent peak of the nitrile rubber (or equivalently, of lowering the temperature of the loss-tangent maximum). One way to do this was to produce samples in which part of the nitrile polymer was replaced by a polymer known to have a lower glass transition temperature. Natural rubber was chosen here as the diluent and samples containing 10 and 20 parts were acquired, molded, and tested. The dynamic moduli of these are given in Fig. 3.
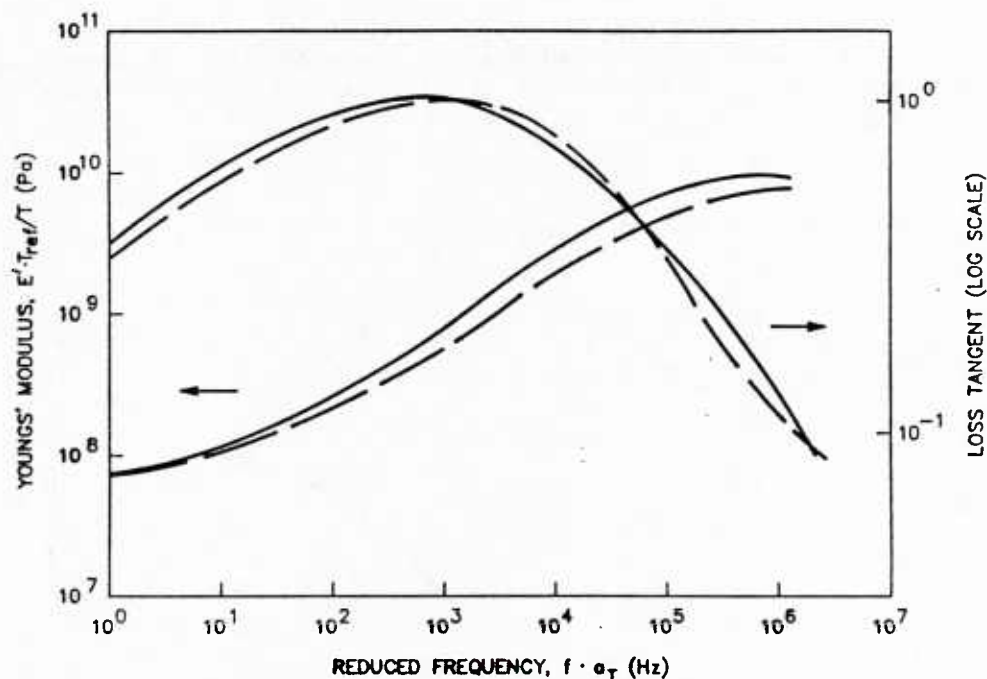
Fig. 3 – Dynamic Young's moduli and loss tangents of a formulation containing 90 parts of 39%-acrylonitrile polymer, 10 parts of natural rubber, and 50 parts of N550 carbon black (————, Arrhenius constant = 7148 cal/mole), and of a formulation containing 80 parts of a 39%-acrylonitrile polymer, 20 parts of natural rubber, and 50 parts of N550 carbon black (_ _ _, Arrhenius constant = 7210 cal/mole).

Careful comparison of the loss tangent curves of these samples to the loss tangent curves of Nitrile 6100 will reveal that the addition of natural rubber did not shift the loss peak to higher frequencies as anticipated. Instead, the loss peaks are broadened and somewhat reduced in amplitude. Rather than the loss tangent peak occurring at a frequency that is a weighted average of the two polymer components as expected, each component is apparently exhibiting its own characteristic loss peak. The peak for the natural rubber polymer is simply unresolved from the peak for the nitrile polymer. From a polymer chemistry perspective, this suggests that the natural rubber is not dissolved in the nitrile rubber but instead is present in the form of finite inhomogeneities. It might be speculated that the cause for the failure of the two polymers to form a homogeneous solution lies in the method of mixing. It is possible that the two polymers have greatly different viscosities as they are mixed simultaneously in the Banbury mixer and that this prevents their being effectively homogenized.

This apparent phase separation means that using another polymer to attempt to shift the loss tangent peak may not be effective. An alternative to this would be to shift the frequency of the loss tangent maximum by changing the copolymer ratio of the nitrile polymer. Such a polymer should be homogeneous. The literature states that reducing the acrylonitrile content lowers the glass transition temperature. The Williams-Landel-Ferry (WLF) relationship suggests that this reduction in the acrylonitrile content would correspondingly raise the frequency of the loss tangent maximum. Thus samples were acquired that were based on copolymers with a range of acrylonitrile contents. Some of

these were also acquired containing several carbon-black loadings. The dynamic Young's moduli for these samples are given in Figs. 4 through 8 below. The samples' compositions are given in the figure captions and are also tabulated in Table 1 which is shown later.
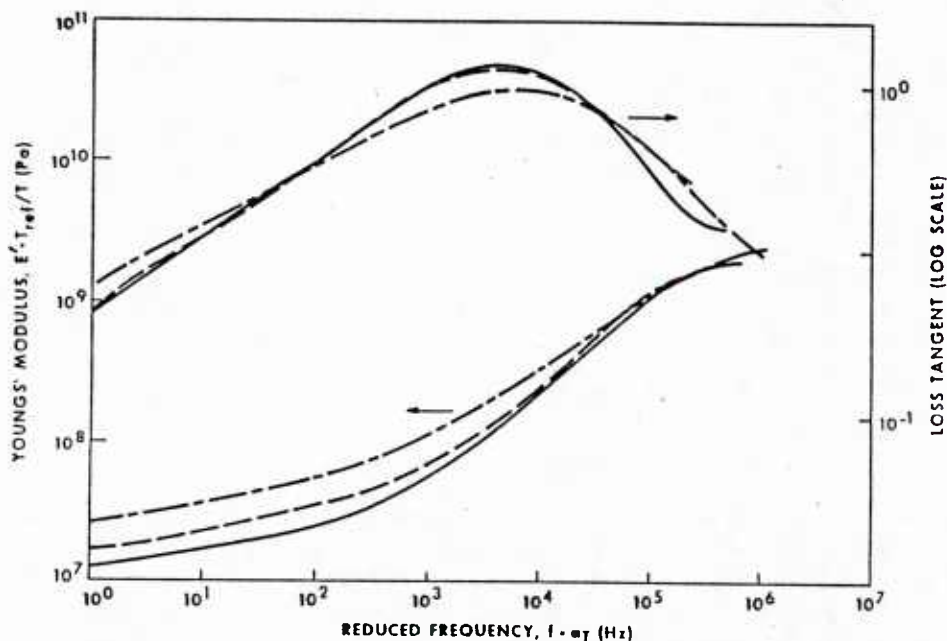

Fig. 4 - Dynamic Young's modulus and loss tangent of a 32.5%-acrylonitrile NBR formulation. _____ 40 phr black, ------ 50 phr black, -.-.-.- 60 phr black. Arrhenius constants are 6190, 6360, and 7225 cal/mole, respectively.
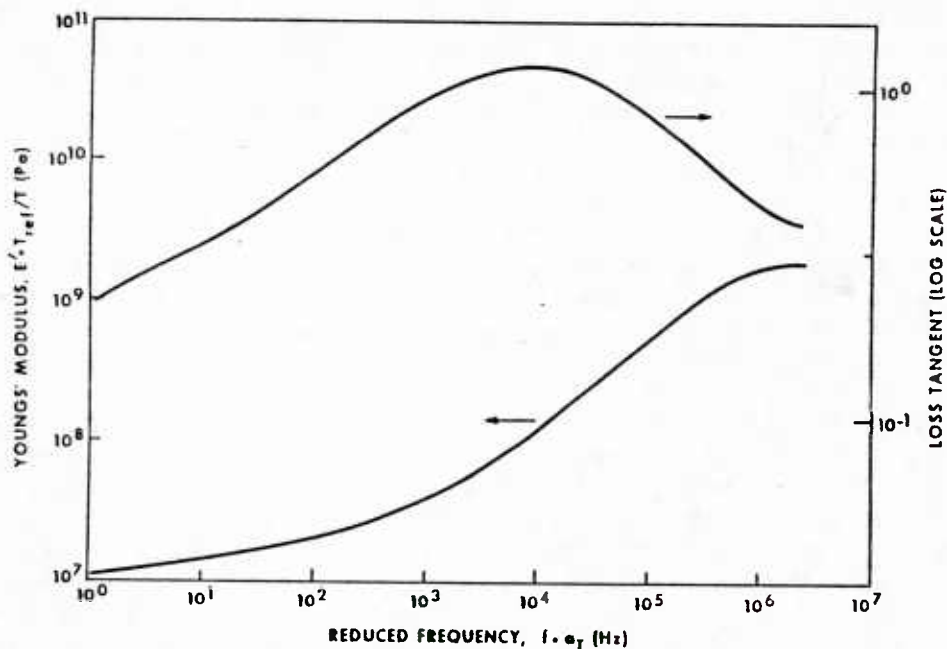

Fig. 5 - Dynamic Young's modulus and loss tangent of a 29.5%-acrylonitrile NBR formulation with 45 phr black. The Arrhenius constant is 6452 cal/mole.
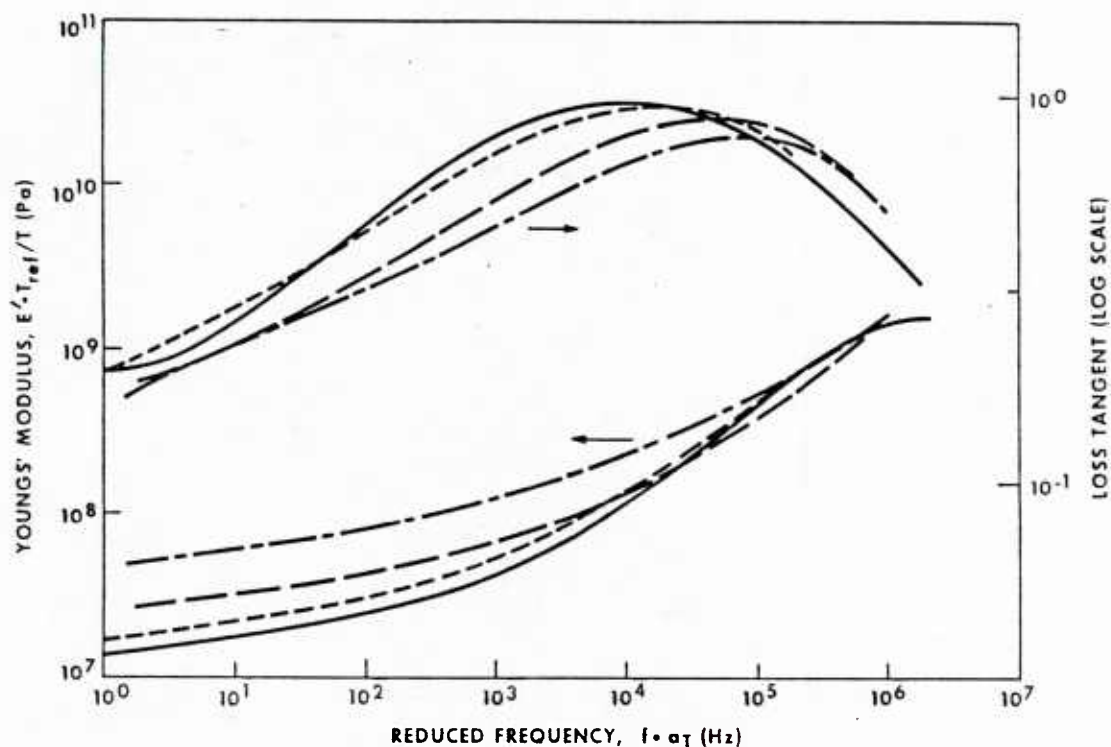
Fig. 6 - Dynamic Young's modulus and loss tangent of a 27%-acrylonitrile NBR formulation. _____ 45 phr black, _ _ _ _ 55 phr black, _ _ _ 65 phr black, — - — 85 parts black. Arrhenius constants are 6292, 6647, 6785, and 7290 cal/mole, respectively.
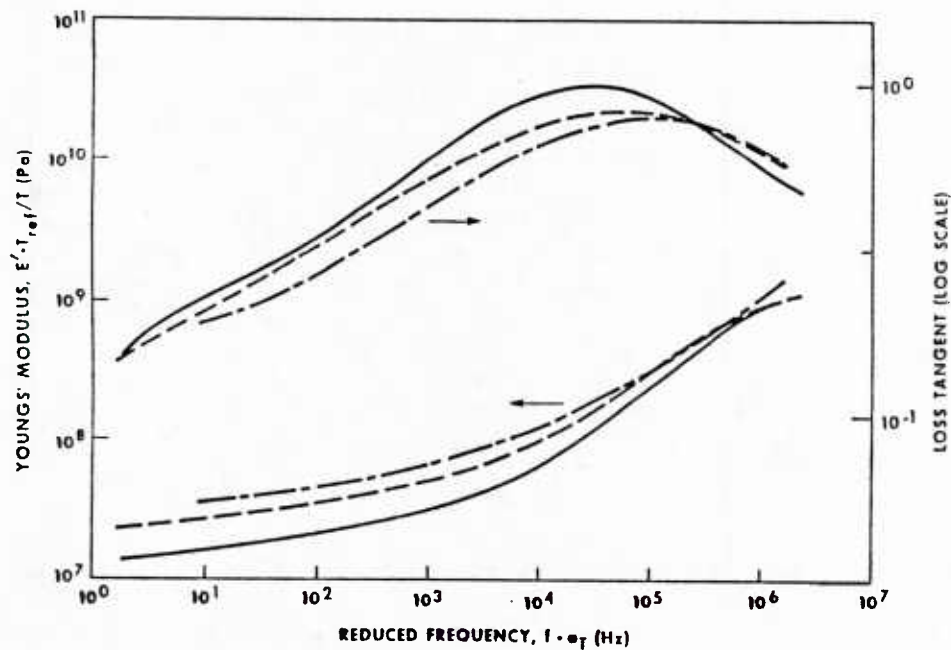

Fig. 7 - Dynamic Young's modulus and loss tangent of a 23.5%-acrylonitrile NBR formulation. _____ 45 phr black, - - - - - 55 phr black, -.-.-.- 75 phr black. Arrhenius constants are 6293, 6594, and 6885 cal/mole, respectively.
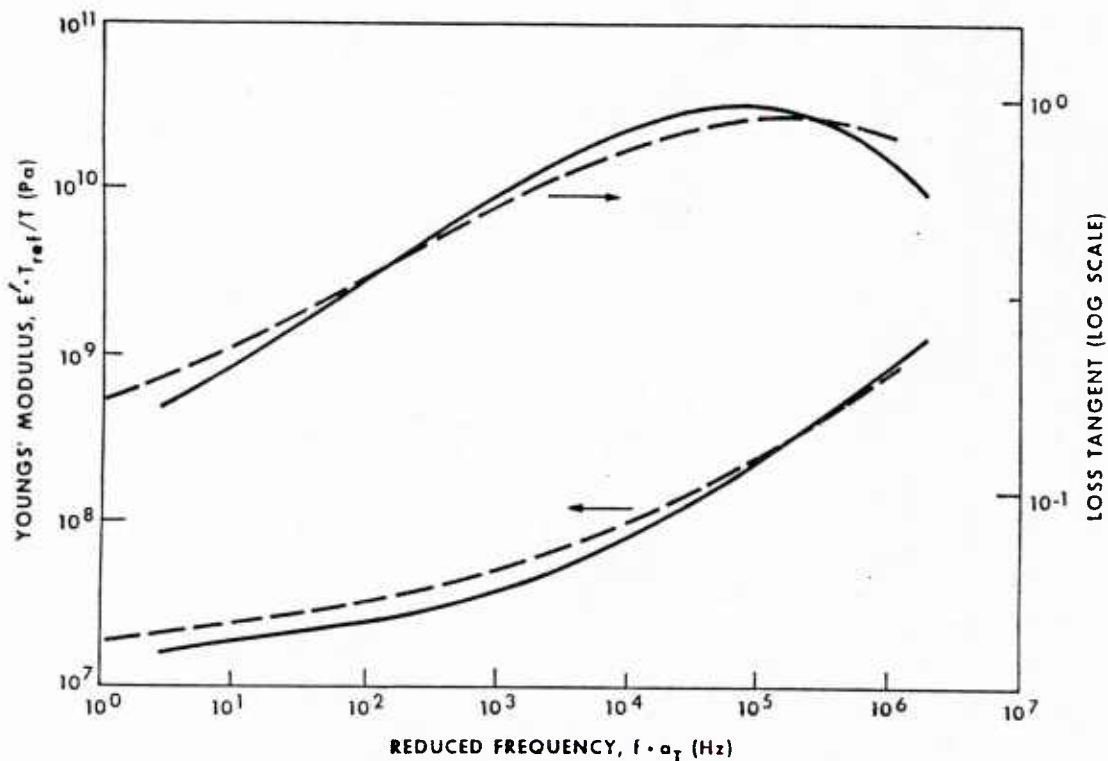
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Fig. 8 – Dynamic Young's modulus and loss tangent of a 32%-acrylonitrile NBR formulation. _____ 45 phr black, ----- 55 phr black. Arrhenius constants are 5984 and 6598 cal/mole, respectively.

These data may be looked at from the perspective of simply trying to match the dynamic modulus of the proprietary butyl rubber system. The samples containing 29.5 or 32.5% acrylonitrile come closest to the target rubber in the frequency of the loss tangent maximum. Furthermore the sample containing 32.5% acrylonitrile and 40 parts of N550 carbon black fairly closely matches the target modulus over the intermediate frequency range. This sample was chosen for further testing and was molded into a TR-317R tail mass assembly (TMA). This assembly was tested on a tail mass dynamic tester (TDYT) [9] and was found to have a stiffness and frequency bandwidth that suggested that it would closely balance the dynamic performance of the transducer. The TMA was assembled into a test TR-317R transducer which was then calibrated. The calibration results showed that the losses of the test transducer were balanced when this TMA was used. Based on these tests, this formulation was specified for use in the TR-317R transducer and was given the designation "Nitrile BLT2." A great deal of subsequent research effort has been done on this formulation as will be described later.

Variation of Loss Tangent Maximum of Nitriles

From a scientific perspective, the data on the several nitrile rubber formulations show some important trends. The values of frequency at which the maximum in loss occurs (at 10°C) for these may be abstracted from the plots of Figs. 1 and 4 through 8. Furthermore, the temperature at which the maximum in loss occurs at a fixed frequency (in this case, 10 kHz) may be calculated from the Arrhenius equation given previously and using the constants given in the figure captions. Table 1 presents these values.

Table 1 - The loss tangent maximum frequency (at 10°C) and temperature (at 10 kHz) of nitriles with several different copolymer acrylonitrile contents and N550 carbon-black loadings.

Percent Acrylonitrile	Carbon Black Loading (phr)	Loss Tangent Maximum		Arrhenius Constant (cal)
		Freq. (10°C)	Temp. (10 kHz)	
39	50	600	26.4	6104
32.5	40	5000	14.0	6190
32.5	45	5000	14.0	6360
32.5	60	6500	12.1	7225
29.5	45	10000	10.0	6452
27	45	10000	10.0	6292
27	55	20000	3.9	6647
27	65	50000	2.0	6785
27	85	80000	0.4	7290
23.5	45	20000	6.2	6293
23.5	55	60000	0.8	6594
23.5	70	110000	-2.5	6385
22	45	80000	-1.8	5904
22	55	150000	-3.8	6508

The trend of loss tangent maximum with acrylonitrile content is evident from Table 1. Furthermore, there also seems to be a regular change in loss tangent maximum with carbon black content. In Fig. 9, the loss tangent maximum frequency as a function of the acrylonitrile content of the polymer is plotted.
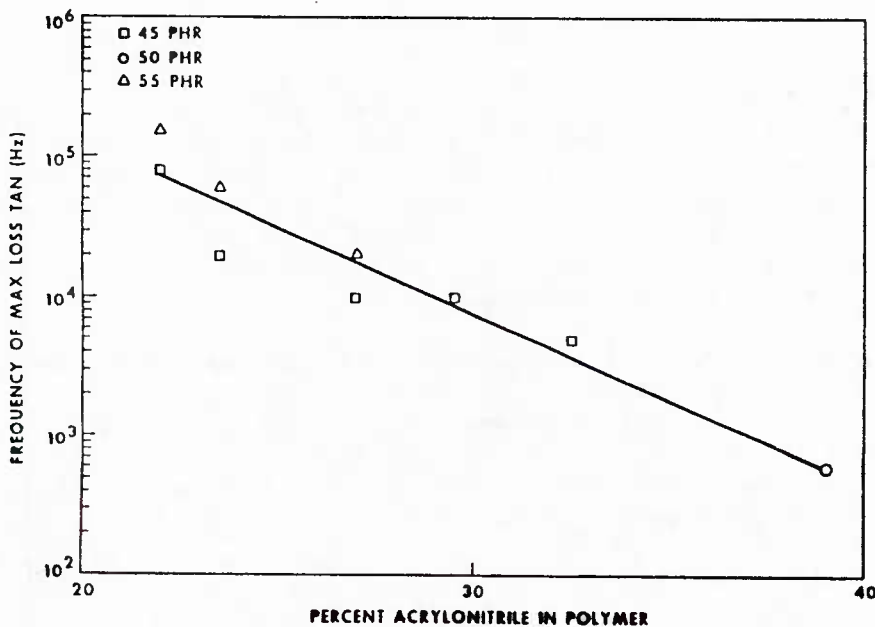

Fig. 9 - Loss tangent maximum frequency (at 15°C) of nitrile rubber formulations as a function of acrylonitrile content of the prepolymer.

The line on this plot is a least-squares fit of the data. The scatter of the data about the line is mostly a result of the additional dependence of the loss tangent maximum on carbon-black loading. This dependence is plotted in Fig. 10 for the cases where multiple carbon-black loadings were available at a given acrylonitrile content. The lines given for each acrylonitrile content are **least squares fits of the data.**
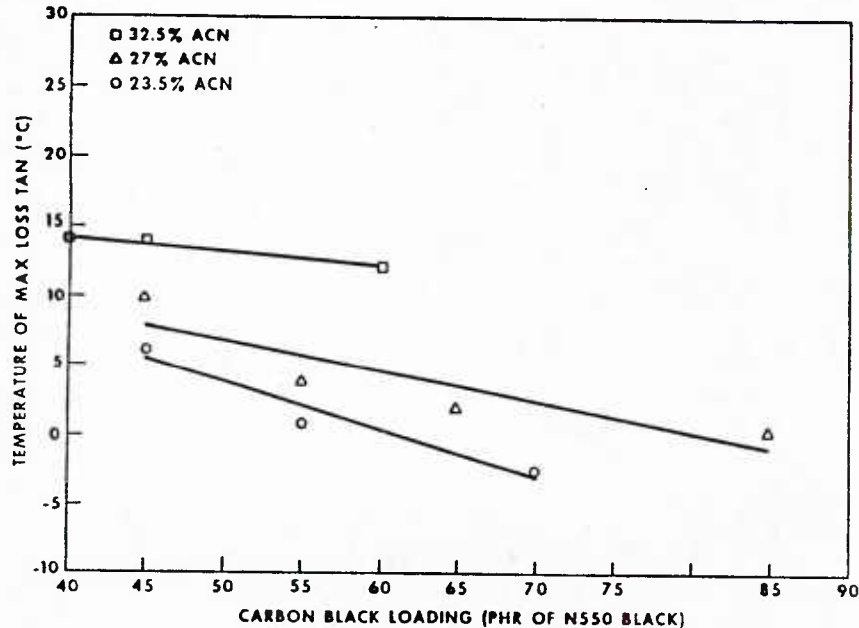

Fig. 10 - Loss tangent maximum frequency (at 10°C) of nitrile rubber formulations as a function of carbon-black loading.

The data of Madigosky et al., which was discussed in the introduction [7], showed a maximum in loss tangent at a frequency that was a factor of 5 to 7 times higher than that shown here. This difference is largely explained by fact that Madigosky's formulations contained significant concentrations of plasticizing ingredients. These plasticizers would indeed be expected to lower the glass-transition temperature or, equivalently, to raise the frequency of the loss tangent maximum.

Vibration damping devices are commonly designed to operate most efficiently at a given frequency. The significance of this data set is that nitrile formulations can now be chosen that exhibit a maximum in loss at any given frequency (from sonic to ultra-sonic) over a limited range of temperatures. Formulations chosen using these data as guides should produce enhanced performance of many vibration damping devices.

Engineering of Tail Mass Assemblies

The early work in developing the TMA damper was clouded by the mystique that had developed around the proprietary butyl formulation which we were replacing. The butyl rubber was said to function only within a very narrow range of shear modulus, or Shore hardness. Consequently, the original specification for the Nitrile BLT2 formulation incorporated very narrow ranges of allowable values for both Shore hardness and acrylonitrile content. An off-specification study was designed and begun wherein these individual

parameters were purposely varied from their specified values. The
concentration of sulfur was also varied since this was perceived as an
important error that might be made in mixing the samples. Samples were
acquired with five different carbon-black loadings, with three acrylonitrile
contents and with three sulfur contents. These were molded into TR-317R TMA's
at the Naval Weapons Support Center in Crane, IN. The TMA's were then tested
in a TDYT at the Naval Oceans System Center in San Diego, CA. Table 2 gives a
summary of the most significant data on the TMA's constructed from these
rubber samples. The current specification for the TR-317R requires the
calculated TMA stiffness to be between 1.9 and 2.2×10^9 N/m and the measured
bandwidth to be between 100 and 230 Hz (all at 23°C).

Table 2 – Summary of results of an off-specification study on Nitrile
BLT molded into TR-317R TMA's.

Sample Number	Off-Specification Ingredient	TMA Average Bandwidth (Hz)	TMA Average Stiffness (100 N/m)
1	Standard BLT2	130	2.15
2	27.8% ACN	103	2.08
3	36.2% ACN	229	2.26
4	20 phr Carbon	78	2.05
5	30 phr Carbon	105	2.05
6	50 phr Carbon	144	2.17
7	60 phr Carbon	140	2.28
8	0.75 phr Sulfur	106	2.07
9	2.50 phr Sulfur	177	2.09

The scatter of the data implies that there are uncontrolled variables in
this experiment. Nevertheless, some trends are clear. The sample with high
acrylonitrile content has a bandwidth that is slightly high. Nevertheless it
seems clear that the permitted range for the acrylonitrile content could be
widened somewhat, perhaps from the present 32.5 ±1.5% to ±2.5%. The series
with increasing carbon-black content shows an almost regular increase in both
stiffness and bandwidth. The measured Shore hardnesses for the series 4, 5,
1, 6, and 7 which contains 20 through 60 phr of carbon black in 10 phr steps,
was 52.6, 57.3, 61.3, 65.0, and 69.3, respectively. The sample with lowest
carbon-black content was low in stiffness while the one with highest carbon-
black content was high in bandwidth. The samples that were 10 phr lower or
higher than the standard 40 phr of carbon black performed acceptably. This
implies that a Shore hardness between 57.3 and 65.0 should be acceptable. A
specification of 61.5 ±3.5 seems adequate from these data. The variation in
sulfur content produced only one significant change. High sulfur content
caused an increase in bandwidth without a concomitant increase in stiffness.
This observation may prove useful in the future if such an increase in
bandwidth is required. All of the details of the work on the off-
specification TMA study are given in a report by Nichols, et al. [9].

Concurrent with the development work on the Nitrile BLT2, research was
carried out on a quality control scheme for this formulation. The approach to
this was to develop methods for the analysis of the components that are in the
uncured rubber. This compositional analysis was to be further supported by
limited physical tests on cured samples of each batch. The compositional
analysis was based on liquid chromatography applied to a filtered solution of
the uncured sample. This served to measure the concentration of each of the

organic additives. Carbon-black analysis was initially done by gravimetric analysis of the filtered residue. This proved to be inadequately precise to control the carbon-black content. A method was subsequently developed based on thermo-gravimetric analysis that showed better precision. Acrylonitrile content is now determined by a Kjeldahl nitrogen analysis applied to a sample which has been extracted to remove other nitrogen-containing additives. The acrylonitrile content of the polymer is calculated from the appropriate gravimetric factor after compensating for the weight of the other additives. This method gives results that are just adequate to control this important factor. Consequently, a method is now being developed in which the acrylonitrile content is determined from the intensity of the nitrile band in the infrared spectrum. This band intensity is normalized to the intensity of a carbon-hydrogen stretching band. This method promises improved precision and it eliminates the assumptions of the Kjeldahl method.

The compositional analysis procedures are supported by tests of the Shore hardness of a cured sample. Hardness is a vital measure of the performance of the rubber batch as was suggested above. Consequently, the Shore hardness measurement is specified as being done on a stand-mounted tester for which both the load and the rate of load application is controlled. The test is made more representative of the rubber batch by averaging 25 measurements made over an area of at least 15×15 cm. Additionally, the cure rate of each batch is monitored by means of a Monsanto 100S rheometer. The details of the quality control scheme now in use are given in a report by Thompson, Quinn and McGee [10].

An additional quality control issue was raised by a user of the Nitrile BLT2 formulation. He wanted to make the rubber in large Banbury masterbatches and then add the TMTM accelerator to batches only as needed. This obviously complicates the compositional analysis quality control because each small batch would have to be tested for TMTM content. The user made the suggestion that the Monsanto 100S rheometer test that is already done might also provide a method of monitoring the TMTM level in the rubber. As a test of this idea, samples were acquired in which the TMTM contents were 0.3 and 0.7 instead of the specified 0.5 phr. The rheometer test on these samples showed that the slope of the increase in shear modulus during the early stages of cure, indeed depends directly on the TMTM content. Unfortunately, this slope was also found to depend on the acrylonitrile content and on other, undetermined variables. Thus this method of specifying TMTM content is not acceptable as a specification test. The issue is of diminished importance since, as explained in the next paragraph, the Nitrile BLT2 does not degenerate on storage. Thus the TMTM component can be added to the batch as it is mixed initially, perhaps on a large mill.

A study was conducted on the aging of Nitrile BLT2 in which fresh samples were stored under a variety of conditions and their condition monitored by means of the Monsanto rheometer. At temperatures up to 40°C, the increase in scorch (or premature vulcanization) was unmeasureable up to several months. Thus, Nitrile BLT2 may easily be stored under ordinary warehouse conditions with only slight regard to its scorch.

Nitrile BLT2 has attractive engineering properties in addition to its dynamic vibration damping. For instance, it is easy to mold under many conditions, and it is easy to bond with both a vulcanizing and a "cold" bond. It has moderately high strength and toughness, and it is relatively high in its resistance to flow under a compressive stress. It has good resistance to oxidation and good resistance to degradation by ozone except where it is

placed under a tensile strain. Because of these attractive properties, Nitrile BLT2 was also selected to be used for the overmolded rubber on the end seal assembly of the TR-317R transducer. Its dynamic lossiness will also serve to dampen any spurious resonances of this assembly.

CONCLUSIONS

A nitrile rubber formulation was designed to match the dynamic properties of a proprietary rubber and was shown to offer at least comparable performance when incorporated into the tail mass assembly of a TR-317R transducer. The study also showed that nitrile rubber formulations offer great design flexibility for other dynamic absorbers in that they can be designed with a maximum in loss at any chosen frequency or temperature.

ACKNOWLEDGMENTS

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